Purpose: An internal Technical System Audit (TSA) is planned on the QA Category A project (QA Track #D-EMMD-0031346) described in the *QAPP: Non-Targeted Analyses of Per- and Polyfluoroalkyl Substances (PFAS) for the New Hampshire Department of Environmental Services (NHDES)* specifically for the extraction of sampling train filters and sorbent media being performed by National Risk Management Research Laboratory/Air and Energy Management Division (NRMRL/AEMD). The audit will follow the checklist provided below. This checklist was prepared using the QAPP referenced above, SW-846 Method 3542 *Extraction of Semivolatile Analytes Collected using Method 0010 (Modified Method 5 Sampling Train),* and ORD PPM 13.4 Quality Assurance/Quality Control Practices for ORD Laboratories Conducting Research.

Personnel: The following persons are scheduled to be directly involved in the TSA.

Name	Responsibility
Libby Nessley (Auditor)	NRMRL/AEMD QA Manager
Dennis Tabor	AEMD/Immediate Office (IO) Chemist
Jeff Ryan	EPA AEMD Principal Investigatory (PI)

Schedule:

- Auditor observed filter/XAD extractions August 27-30, 2018
- Checklist completed with D. Tabor on September 6, 2018

	Requirement	Compliance			Reference:	Comments/Objective Evidence
		Y	N	NA		
1.0	**General**					
	Were triplicate MM5 train samples, including inlet filter, XAD-s sorbents, and impinger fractions taken from 4 towers at the Saint-Gobain facility submitted to the laboratory for extraction?	✓			QAPP B2	Optimal extraction procedure is currently still under development. One set of filters and XAD were extracted at NERL's request to have a general conc range of target compounds. Recovery of spikes needs improvement.
	Was one field biased blank train received for extraction?	✓			QAPP B2	
	Were samples stored at 4 °C between the time of sampling and extraction?	✓			M3542/6.1	
	Were extractions stored in a refrigerator/freezer at least less than 4 °C?	✓			QAPP B3	
	Did laboratory staff use the assigned sample IDs defined by the field collectors?	✓			QAPP B3	
	Is glassware thoroughly cleaned prior to use by: • Washing in detergent/hot waste/rinse (tap & distilled)? • Baked at 400 C for at least an hour? • Rinsed methanol (X3) & MeCl (X3)?	✓				

Additional Notes:

7 components from each MM5 train for analysis: filter, XAD, methanol rinse, 3 impinger rinses & back filter. Audit only covers extraction of the filter and XAD components from one set of samples.

	Requirement	Compliance			Reference:	Comments/Objective Evidence
		Y	N	NA		
2.0	**Standards & Spike Solution Preparations**					
	Is certification of concentrations of primary standards available?	✓			ORD PPM 13.4	
	Is preparation of stock solutions and spiking solutions adequately documented?	✓			ORD PPM 13.4	Standards Notebook #188
Additional Notes						
3.0	**Quality Control**					
	Are surrogates for compounds of interest spiked into the component prior to extraction?	✓			M3542/5.8	
	Were samples extracted within 14 days of collection?		✓		M3542/6.2	QAPP indicates no hold times associated with these samples.
	Is a method blank extracted and concentrated using the same procedures as the corresponding sample matrix?		✓		M3542/8.2	
	Is a method spike extracted and analyzed?		✓		M3542/8.3	NOTE-numerous method spikes have been performed during method development to determine extraction procedure, which is still under development.
Additional Notes						

	Requirement	Compliance			Reference:	Comments/Objective Evidence
		Y	N	NA		
4.0 Procedure						
Particulate matter filter and front half rinse (3542/7.2.1)						
	Are PTFE boiling chips used in the bottom of the round bottom flask?		✓		M3542/7.2.1.1	Carbide boiling chips are being used. No PTFE being used to reduce risk of contamination.
	Is an aliquot of surrogate spiking solution added directly to the top of filter?		✓		M3542/7.2.1.2	Solution is not added directly to the top of the filter. Added to filter in the soxhlet.
	Is the filter placed in the soxhlet extraction thimble in such a way that the filter is completely submerged in solvent with each cycle of the extraction?	✓			M3542/7.2.1.3	
	Is glass wool placed on top of the filter to keep it in place?		✓		M3542/7.2.1.3	Not needed.
	Is filter container rinsed (X3) with solvent in to the soxhlet?		✓		M3542/7.2.1.3	Filters were received in plastic petri dishes. Decision was made not to rinse to avoid risk of contamination.
	Is a front half rinse associated with the filter?	✓			M3542/7.2.1.4	Will be analyzed as a separate sample.
	Is front half rinse extracted with the filter?		✓		M3542/7.2.1.4	As above.
	Is sample allowed to extract for 18 hours such that the sample cycles approximately once every thirty minutes?	✓			M3542/7.2.1.7	
Front half rinse (3542/7.2.2)						
	Is filtered front half rinse transferred to a separatory funnel by rinsing the sample container at least 3 times with solvent?			✓	M3542/7.2.2.1	Not extracting front half rinse with the filter.
Filter & front half rinse concentration (3542/7.2.3)						
	Are extracts from the filter and front half rinse funneled through glass wool and sodium sulfate in to a K-D flask?		✓		M3542/7.2.3.4	No necessary with methanol as solvent.

	Requirement	Compliance			Reference:	Comments/Objective Evidence
		Y	N	NA		
	Is K-D apparatus placed on a hot water bath (80-85 C) and concentration completed in 20-30 minutes?		✓		M3542/7.2.3.5	No water bath. This temp is for MeCl2 solvent. We are using methanol. Down to 100 mL
	Is final concentration performed by blowing the surface of the solvent with a gentle stream of nitrogen to the final extract volume?	✓			M3542/7.2.3.6	Turbovap to 10 mL
XAD extraction (3542/7.4)						
	Is XAD transferred to an extraction thimble along with any glass wool and trap and glass joints rinsed with solvent in to the extraction vessel?		✓		M3542/7.4.2	Does not include rinses.
	Is an aliquot of spiking solution added to the XAD?	✓			M3542/7.4.3	Pre-extraction standard (50 ng)
	Are the back-half rinses added to the XAD extraction?		✓		M3542/7.4.4	Separate sample.
	Is sample extracted for at least 18 hours cycling once every 25-30 minutes?	✓			M3542/7.4.7	

Additional Notes:

Majority of deviations from Method 3542 are related to the primary deviation which was a solvent change from methylene chloride to methanol. Methanol is more compatible solvent for the LC/MS analysis planned for these extracts. There is still method development work needed to optimize extraction procedure for methanol to improve recoveries.